(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
13 May 2004 (13.05.2004)

PCT

(10) International Publication Number
WO 2004/040033 A1

(51) International Patent Classification[7]: C23C 8/22

(21) International Application Number: PCT/PL2003/000065

(22) International Filing Date: 2 July 2003 (02.07.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
356921 31 October 2002 (31.10.2002) PL

(71) Applicants (*for all designated States except US*): SECO/WARWICK SP. Z O.O. [PL/PL]; ul. Sobieskiego 8, PL-66-200 Swiebodzin (PL). POLITECHNIKA LÓKZKA [PL/PL]; ul. Stefanowskiego 1/15, PL-90-924 Lódz (PL).

(72) Inventors; and
(75) Inventors/Applicants (*for US only*): KULA, Piotr [PL/PL]; ul. Chochola 43, PL-91-230 Lódz (PL). OLEJNIK, Józef [PL/PL]; Osiedle Zaków 61, PL-66-200 Swiebodzin (PL). HEILMAN, Paul [DE/DE]; Tiedstrasse 8, 63469 Maintul (DE).

(74) Agent: NISZTUK, Helena; Lubuski Klub Techniki i Racjonalizacji, ul. Niecala 2F, PL-65-245 Zielona Góra (PL).

(81) Designated State (*national*): US.

(84) Designated States (*regional*): European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR).

Published:
— *with international search report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: METHOD FOR UNDER-PRESSURE CARBURIZING OF STEEL WORKPIECES

(57) Abstract: The subject of this invention relates to method carburizing of steel products, mainly parts of machines, vehicles and every mechanical apparatus, in vacuum furnaces under reduced pressure and elevated temperature. The method of under-pressure carburizing of steel workpieces according to present invention relates to introduction of active nitrogen carrier during heating up of the load. Introduction of the active nitrogen carrier is terminated when the load reaches temperature required to start carburizing process; from this temperature the carbon carrier is added. Pressure in the furnace chamber during continuous or pulse introduction of the active nitrogen carrier should be maintained within the range from 1 to 500 mbar.

WO 2004/040033 A1

Method for under-pressure carburizing of steel workpieces

The object of this invention relates to the method for carburizing of steel products, mainly parts of machines, vehicles and all types of mechanical apparatuses, in vacuum furnaces under reduced pressure and elevated temperature.

A method for carburizing of products made of steel in a furnace chamber is known from the US Patent 6,187,111. In this method, vacuum in the range of 1 to 10 hPa is generated and the temperature of the carburizing process is maintained between 900°C and 1100°C. The carbon carrier there is gaseous ethylene. Another US Patent, 5,205,873, describes the carburizing process carried out under low pressure in a furnace chamber heated up to temperatures between 820°C and 1100°C. This process starts in a chamber where an initial vacuum up to 10^{-1} hPa was generated to remove the air. Then, after backfill of the chamber with pure nitrogen, workpieces to be carburized are placed into it. In the loaded chamber, a vacuum in the range of 10^{-2} hPa is generated and the charge is heated up to the austenitizing temperature and this temperature is maintained until the temperatures across the workpiece are equalised; afterwards the furnace chamber is backfilled with hydrogen up to 500 hPa. Then ethylene as the carbon carrier is introduced under the pressure from 10 to 100 hPa and a gas mixture consisting of hydrogen and ethylene is created, in which the ethylene content ranges from 2% to 60% of the gas mixture by volume.

Also the US Patent 5,702,540, describes the method of carburizing, according to which the charge is pre-heated under vacuum and gaseous unsaturated aliphatic hydrocarbons are used as the carbon carrier. This method can also be applied for carbonitriding, where together with the carbon carrier an active nitrogen carrier is introduced to the furnace chamber.

The method for under-pressure carburizing of steel workpieces according to the present invention consists in the introduction of an active nitrogen carrier during heating up of the charge, preferably after the temperature of 400°C is reached. The introduction of the active nitrogen carrier is terminated when the charge reaches the temperature required to start the carburizing process; as soon as this temperature is reached the carbon carrier is added. The pressure in the furnace chamber during a continuous or pulse introduction of the active nitrogen carrier should be maintained within the range of 1 to 500 mbar.

The most preferable and beneficial effects are obtained when the active nitrogen carrier is ammonia and the pressure during its introduction is maintained within the range of 1 to 50 mbar.

The method according to the present invention is distinguished by a possibility of an effective application of the upper range of carburizing temperatures due to restraining the growth of austenite grains as a result of initial saturation of the surface area with nitrogen and in consequence the process is significantly accelerated.

One of possible implementations of the method for under-pressure carburizing of steel workpieces according to the present invention is illustrated by the following examples:

Example 1

A furnace chamber of the size 200x200x400 mm was loaded with workpieces made of low carbon steel grades C15, 16CrMn5 and 17CrNiMo. The total surface area of the charge was 0.4 m^2. After pre-heating under vacuum up to 400°C ammonia was introduced to the furnace chamber interior with a constant flow rate of 50 l/hr. The process atmosphere was maintained under a constant pressure of 5 mbar. When steel workpieces had reached the temperature of 950°C, the introduction of ammonia was interrupted, and carburizing atmosphere was introduced for twenty minutes and a constant temperature of the vacuum furnace chamber was maintained; the atmosphere was made up of the carbon carrier in the

form of a mixture of ethylene and acetylene in the volume ratio 1, mixed with hydrogen in the volume ratio 1,17, introduced with a constant flow rate 190 l/hr and thus generating pressure pulse in the furnace chamber within the range of 3 to 8 mbar. For the next 8 minutes steel workpieces were heated under vacuum at the temperature of 950°C and then slowly cooled under vacuum down to the ambient temperature. On individual steel workpieces carburized layers were produced with the following performance.

Steel grade	Surface carbon concentration [%]	Case depth to limit structure – 50% perlite + 50% austenite [mm]	Original grain size [mm]
C15	0.65	0.40 ± 0.005	40% -0.008 60% -0.011
16CrMn5	0,71	0,46 ± 0,005	50%-0,011 50%-0,013
17CrNiMo	0,72	0,47 ± 0,005	70%-0,011 30%-0,016

The surface of all workpieces after carburizing was clean and bright without any evidence of soot and tar.

Example 2

A furnace chamber of the size 200x200x400 mm was loaded with workpieces made of low carbon steel grades 16CrMn5 and 17CrNiMo. The total surface area of the load was 0.4 m^2. After pre-heating under vacuum up to 400°C ammonia was introduced to the furnace chamber interior with a constant flow rate of 50 l/hr. The process atmosphere was maintained under a constant pressure of 5 mbar. When steel workpieces had reached the temperature of 950°C, the introduction of ammonia was interrupted, and carburizing atmosphere was introduced for twenty minutes and a constant temperature of the vacuum furnace chamber was maintained; the atmosphere was made up of the carbon carrier in the form of a

mixture of ethylene and acetylene in the volume ratio 1, mixed with hydrogen in the volume ratio 1,17 introduced with a constant flow rate 190 l/hr and thus generating pressure pulse in the furnace chamber within the range of 3 to 8 mbar. For the next 20 minutes steel workpieces were heated under vacuum at the temperature of 950°C and then fast cooled down to the ambient temperature under nitrogen at the pressure increased up to 6 bar. On individual steel workpieces carburized layers were produced with the following performance.

Steel grade	Surface hardness [HV_{01}]	Case depth to limit hardness 500 HV_{01}
16CrMn5	744	0,48 ± 0,005
17CrNiMo	820	0,49 ± 0,005

The surface of all workpieces after carburizing was clean and bright without any evidence of soot and tar.

Example 3

A furnace chamber of the size 200x200x400 mm was loaded with workpieces made of low carbon steel grades C15, 16CrMn5 and 17CrNiMo. The total surface area of the load was 0.4 m^2. After pre-heating under vacuum up to 400°C ammonia was introduced to the furnace chamber interior with a constant flow rate of 50 l/hr. The process atmosphere was maintained under a constant pressure of 5 mbar. When steel workpieces had reached the temperature of 1000°C, the introduction of ammonia was interrupted, and carburizing atmosphere was introduced for twenty minutes and a constant temperature of the vacuum furnace chamber was maintained; the atmosphere was made up of the carbon carrier in the form of a mixture of ethylene and acetylene in the volume ratio 1, mixed with hydrogen in the volume ratio 1,17 introduced with a constant flow rate 270 l/hr and thus generating pressure pulse in the furnace chamber within the range of 3 to 8 mbar. For the next five minutes steel workpieces were heated under vacuum at the temperature of 1000°C and then slowly cooled under vacuum down to the ambient

temperature. On individual steel workpieces carburized layers were produced with the following performance.

Steel grade	Surface carbon concentration [%]	Case depth to limit structure – 50% perlite + 50% austenite [mm]	Original grain size [mm]
C15	0.66	0.52 ± 0.005	70% -0.011 30% -0.013
16CrMn5	0,70	0,58 ± 0,005	50%-0,013 50%-0,016
17CrNiMo	0,70	0,59 ± 0,005	60%-0,013 40%-0,016

The surface of all workpieces after carburizing was clean and bright without any evidence of soot and tar.

CLAIMS:

1. The method of under-pressure carburizing of steel workpieces with the introduction of the active nitrogen carrier to the vacuum furnace chamber is **characterized in that** the active nitrogen carrier is introduced during pre-heating of the charge until the charge reaches the carburizing temperature and the pressure in the furnace chamber is maintained within the range of 1 to 500 mbar.
2. The method according to claim 1 **is characterized in that** the said active nitrogen carrier can be introduced to the furnace chamber in a continuous or pulse manner.
3. The method according to claim 1 **is characterized in that** it is most beneficial and preferable if the pressure during the introduction of the said active nitrogen carrier is maintained within the range of 1 to 50 mbar.
4. The method according to claim 1 **is characterized in that** it is beneficial and preferable if the introduction of the said active nitrogen carrier starts once the temperature of the charge reaches 400°C.
5. The method according to claim 1 **is characterized in that** it is most beneficial and preferable if the said active nitrogen carrier is ammonia